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"1-Nitro-4,3-Naphthalenedisulfonic Acid and 2-Nitro-4, 8-Naphthalenedisulfonic Acid," A.A. Chuksanova, E.V. Sokolova, Sci Res Inst of Org Intermediate Products and Dyestuffs imeni K. Ye. Voroshilov, Moscow

"Zhur Prikład Khimii" Vol 18, 1945, pp 55-8

Na salt of nitro C-acid (3-nitro-1,5-naphthalenedisulfonic acid) heated with four parts of PCl_5 for 5 hours on a steam bath gave, on pouring into cold water and crystallization from benzene, the corresponding bis(sulfonyl chloride), melts at 158°; similarly, nitro-S-acid (4-nitro isomer) gave the bis(sulfonyl chloride), melts at 177°. Nitration of 1,5-naphthalenedisulfonic acid with 63% HNO_3 in H_2SO_4 gave both the 3- and the 4-nitro isomers in following ratios: in 94% H_2SO_4, 33% 3-nitro and 60% 4-nitro acid at 20-5°, 39% and 59% at 5-10°; in 97% H_2SO_4 at 15-20°, 42% and 50%, respectively.

"Determination of Small Quantities of Monophenols in the Presence of Polyphenols and Quinone," I.I. Ioffe, E.V. Sokolova, Sci Res Inst of Org Intermediate Products and Dyestuffs imeni K. Ye. Voroshilov, Moscow

"Zhur. Prikład Khimii" Vol 18, 1945, pp 273-7

Small quantities of monophenols can be determined in the presence of polyphenols and quinone by oxidation of the latter with H_2O_2. Treat 10 ml of the test solution, containing about 0.0002 mol/l, with 1 ml of 25-30% H_2O_2 and 5 ml 20% NH_4OH; after 2-3 hours remove excess H_2O_2 by adding MnO_2, neutralize the filtrate with HCl, and

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treat with diazosulfanilic acid solution in the presence of Na_2CO_3 and NH_4OH at 0-5°, and measure the color in a photometer.

"Carbazole and Its Derivatives: IV. Selective Action of Acylating Substances on N-(Hydroxymethyl) Carbazole," K. G. Mizuch, Ts. M. Gel'fer, Sci Res Inst of Org Intermediate Products and Dyestuffs imeni K. Ye. Voroshilov, Moscow

"Zhur Prikład Khimii" Vol 19, 1946, pp 939-44

9-(Hydroxymethyl) carbazole (I), dry C_5H_5N, and Ac_2O heated 30 minutes at 100° give 96.8% crude or 65-70% purified 9-(acetoxymethyl) carbazole, melting at 81-2°, which, like its analogs, decomposes when heated and hydrolyzes very easily. The analogous reaction with Bz_2O gives 96.2% 9-(benzoxymethyl) carbazole, melting at 99.8-100.8°. When the reactions are carried out with AcCl or BzCl instead of the anhydrides, a small amount of solid mixture of 9,9-dicarbazolylmethane, melting at 312-13°, and the acyl derivative of I are formed. When the filtrate from this mixture is allowed to stand, 68% I slowly separates. Warming increases the rate of separation. The chief reaction product in this case is an unstable quaternary [redacted] ammonium compound which decomposes, regenerating I. 3-Nitrocarbazole heated with K_2CO_3, EtOH, and CH_2O gives 88% 9-(hydroxymethyl)-3-nitrocarbazole, melting at 183-3.4°. With either Ac_2O or AcCl this gives 93-5% 9-(acetoxymethyl)-3-nitrocarbazole, melting at 157-7.8°, and with Bz_2O or BzCl it gives 97-9% of the benzoxy derivatives, melting at 163-4°. An analogous reaction gives 9-(hydroxymethyl)-3-benzoylcarbazole, melting at 154.5-5.5°, which with Ac_2O gives 91% and with AcCl 47% of the acetoxy derivative, melting at 158-9°. Thus the course of the reaction depends on the acetylating and the acetylated compound.

"Carbazole and Its Derivatives: III. Action of Dialkylsulfates on Sodium 3-Carbazolesulfonate," K. G. Mizuch, Ts. M. Gel'fer, Sci Res Inst of Org Intermediate Products and Dyestuffs imeni K. Ye. Voroshilov, Moscow

"Zhur Prikład Khimii" Vol 17, 1947, pp 823-8

Pure neutral R_2SO_4 do not alkylate or sulfonate Na 3-carbazolesulfonate (I). I (8.07 g), 22.7 g Me_2SO_4, and 20 cc MePh gently refluxed 3.5 hours yielded 99% unchanged I; similar results were obtained with Et_2SO_4. When 8.07 g I, 22.7 g Me_2SO_4, 20 cc MePh, and 0.54 g H_2O were treated as above, the reaction mass was deep violet; after evaporation of the PhMe and Me_2SO_4, the residue, diluted with H_2O and neutralized with Na_2CO_3, gave 6.55 g of a mixture, melting at 214-19°, of mixed di-Me esters of carbazole-disulfonic acids; no I was found. The crude di-Me esters were hydrolyzed by alcohol NaOH, freed of EtOH, and the products were heated in sealed tubes with 7%

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HCl 8 hours to 165-70°; crystallization of the resulting mixture from PhMe gave carbazole and 9-methylcarbazole in the ratio of 2:1. It was impossible to completely identify the components of the Me_2 (or Et_2) ester mixture since crystallization led to hydrolysis and loss of all but esters of 3, 6-carbazoledisulfonic acid, which are colorless solids, insoluble in H_2O, do not react with 4-OHC_6H_4OH in H_2SO_4, and decompose on heating: di-Me ester, long needles (from dry MeOH), decompose 235-6°; di-Et ester, needles decompose 211-12° (slow heating), or 222-3° (rapid heating). Addition of increasingly greater amounts of H_2O or H_2SO_4 favors the formation of such ester mixtures. Similarly the use of commercial H_2SO_4, containing acid, leads to such ester mixtures. Similar promotion of the reaction was obtained with Et_2SO_4 which was heated 3 hours to 170° before the reaction; the more stable Me_2SO_4 did not show an effect. The mechanism of the N-alkylation is not clear, but the sulfonating action is caused by the monoalkyl sulfates produced by the action of either H_2O or H_2SO_4, on R_2SO_4.

"Alkylation of Organomagnesium Compounds by Esters of Carboxylic Acids," K. G. Midzuch, Sci Res Inst of Org Intermediate Products and Dyestuffs imeni K. Ye. Voroshilov, Moscow

"Zhur Obshch Khimii" Vol 16, 1946, pp 1471-4

To MeMgI (from 1.8 g Mg and 8.5 g MeI) in 15 cc Et_2O was added at 0° 6 g 9-(acetoxymethyl) carbazole (I); after standing one hour with cooling the mixture was hydrolyzed to yield 73.9% 9-ethylcarbazole, melting at 67.4-7.8° (from EtOH); picrate melting at 101.2-2°. Similarly, EtMgBr gave 67% 9-propylcarbazole, melting at 48.2-9°; picrate melting at 98-8.5°; PhMgBr gave 75.9% 9-benzylcarbazole, melting at 118-18.8° (from EtOH). PhMgBr (from 4.75 g PhBr) cooled to 0° and treated with 7.62 g 9-(benzoxymethyl) carbazole in 80 cc Et_2O gave 9-benzyl-carbazole and BzOH (1.4 g). The crude Mg derivative of $PhNH_2$ (from 1.8 g Mg, 8.6 g MeI, and 5.6 g $PhNH_2$) in 40 cc Et_2O with 6 g I in 70 cc Et_2O at 15-24° heated to 35° with stirring 2 hours yielded, after the usual hydrolysis, 0.8 g di-9-carbazolylmethane (II), melting at 313-14° (from benzene), and 2.05 g N-(9-carbazolylmethyl) aniline, melting at 147-8.5° (from EtOH); the first compound was prepared in 72.5% yield by addition of 6 g I to 9-carbazolylmagnesium iodide (from 1.8 g Mg, 8.5 g MeI, and 10 g carbazole) at 15-20° when, after the usual working-up there was obtained II, melting at 313-14° (from PhMe). This product is different from that obtained by Silvermacher and Loeb; the latter is the 3,3'-isomer.

"Kinetics of the Reaction Between Allyl Chloride and Sulfuric Acid," I. I. Ioffe, L. M. Morozovskaya, Sci Res Inst of Org Intermediates Products and Dyestuffs imeni K. Ye. Voroshilov, Moscow

"Zhur Fiz Khimii" Vol 21, 1947, pp 545-8

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The rate v of adsorption of gaseous C_3H_5Cl and propylene by 82-88% H_2SO_4 without any stirring or shaking is proportional to the partial pressure of the gas at any given moment; the initial pressure was 80 mm Hg for C_3H_5Cl and 100 mm Hg for C_3H_6, the other gas being air. The v for C_3H_6 is 5-8 times that for C_3H_5Cl. When liquid C_3H_5Cl and C_3H_6 are shaken with 76-86% H_2SO_4, the v is independent of the amount dissolved and increases three times when the temperature rises from 35° to 45°. The v of C_3H_6 is 15 times that of C_3H_5Cl.

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